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4D Study of Grain Growth in Armco Iron Using Laboratory X-ray Diffraction Contrast Tomography

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Abstract. Using a novel laboratory diffraction contrast tomography (LabDCT) technique, a non-destructive 4D study was conducted to investigate the evolution in 3D of the grain structure during grain growth in an Armco iron sample. The 3D grain morphology and the crystallographic orientations of more than 300 grains were determined at three temporal states during annealing. The correlation between growth of grains and grain orientation is explored. The results demonstrate the capability of the LabDCT technique to allow detailed studies of grain growth, and thereby provide the necessary 4D experimental evidence required for further understanding of grain growth.

1. Introduction

The majority of metallic engineering materials are polycrystalline and their properties are strongly dependent on thermomechanical processing such as plastic deformation and annealing. During annealing grain growth can occur, with some of the grains growing at the expense of others. Grain growth is important since it affects the distribution of boundaries and sizes of the grains in the microstructure, and this is intimately linked to the properties of a material.

The kinetics of grain growth has been a main focus of several studies, investigating factors such as the topology of the grains and the grain boundary properties [e.g. 1-3]. While considerable efforts have been made to model the grain growth process, there have been considerably fewer efforts to provide experimental verification of these models [4, 5]. Essentially, grain growth is a 3D structural evolution process, so that neither 2D examination on section, nor 3D measurements based on serial sectioning are capable of providing direct 4D experimental evidence to validate the theoretical models. With the development during the last two decades of novel X-ray-based diffraction-imaging techniques, non-destructive 3D mapping of grain structures is now possible. Experiments using high-energy synchrotron X-ray beams to study the 3D grain structure evolution during annealing have been reported [5-9], however, the experiments dedicated to grain growth studies are still small in number due to the limited access to international synchrotron facilities.



The novel laboratory-based X-ray diffraction contrast tomography system (LabDCT), operating on a commercially available X-ray microscope (XRM), enables the wider accessibility and routine use of non-destructive, time-evolution experiments, thereby allowing the necessary large number of experiments required for grain growth studies to be carried out [10, 11]. In this work, preliminary results from a 4D study of the grain growth behavior in an Armco iron sample using LabDCT are presented. Essential information regarding the microstructural evolution, including both morphology and crystallographic orientation of the grains, is revealed. Analysis of the grain growth kinetics in correlation with grain orientation is presented to demonstrate the potential of LabDCT in advancing the understanding of grain growth.

2. Experimental method

The material used in the present study is Armco iron with very low carbon content. The initial material was obtained from a hot-rolled bar. A pre-treatment was carried out to refine the grain size, consisting of 75% cold rolling reduction in thickness and subsequent annealing at 880 °C for 5 days. The average starting grain size after pre-treatment was approximately 75 μm . The microstructure consisted of only polygonal ferritic grains without presence of carbides. A cylindrical sample with diameter of 1 mm was prepared by electrical discharge machining for the LabDCT grain growth studies. The axis of the cylinder was taken along the normal direction (ND) of the initial hot-rolled bar.

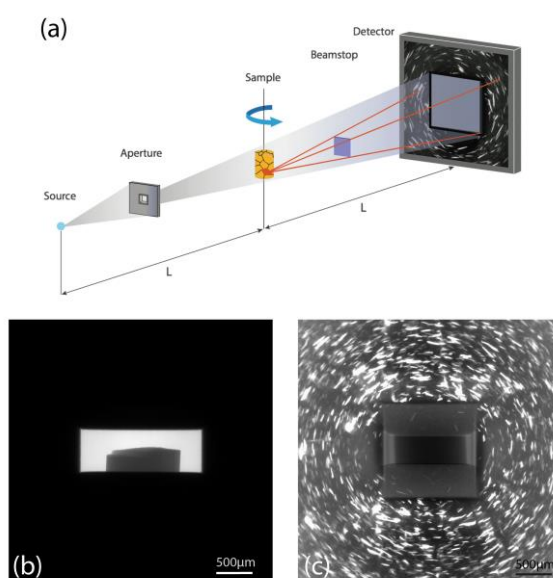


Figure 1. (a) Schematic showing the experimental setup of LabDCT in the laboratory X-ray microscope [10]. (b) Example absorption contrast projection with the direct beam illuminating the top part of the Armco iron sample. (c) Example projection showing diffraction spots from grains within the scanned volume of the sample.

The LabDCT characterization was conducted on a commercially available ZEISS Xradia 520 Versa X-ray Microscope (with LabDCT module). Figure 1(a) schematically illustrates the working principles of LabDCT. The instrument uses a polychromatic, divergent X-ray beam, instead of the parallel monochromatic beam typically used in the synchrotron X-ray DCT technique. An aperture is placed between the source and the sample, to constrain the incoming X-ray beam and to illuminate the sample only in the center region of the detector, as seen in the example shown in figure 1(b). For the DCT scan, a beam-stop is used to block the transmitted X-rays to increase the sensitivity of the diffraction measurements. A high-resolution detector is placed at the Laue focal plane with equal source-detector distance. In this arrangement the crystal grains fulfilling the Bragg condition focus the divergent X-ray

beams into a line in the diffraction pattern. Figure 1(c) shows an example of the diffraction spots of the current sample at a single rotation position. As the sample rotates, a specified number of DCT scan projections can be collected through a 360-degree rotation. The collected data were processed and reconstructed with the GrainMapper3D™ analysis package developed by Xnovo Technology ApS. The reconstruction generates a 3D map of the grains in the scanned volume, including both the crystallographic orientation and morphology of the grains. In the present work, the volumes of interest in the as-treated sample were first scanned acquiring 181 DCT projections over a 360-degree rotation. Annealing treatments of 18 hrs at 880 °C and then 21 hrs at 880 °C were subsequently carried out using an external furnace. After each annealing treatment, the volumes of interest in the sample was then scanned with the same experimental settings to track the microstructural evolution. At each state two volumes with a specified overlapping distance in height were scanned and subsequently stitched together. The three temporal states are named as t_0 , t_1 and t_2 in the following text.

3. Results and Discussion

Figure 2(a) shows the reconstructed 3D grain maps of the same volume at the three temporal states. It should be noted that these 3D grain maps are preliminary results implementing a new algorithm for shape reconstruction and a full validation is still ongoing. The color chosen is based on the crystallographic orientation of the grains with respect to the sample rotation axis, which is the RD of the initial hot-rolled bar. Each voxel in the reconstructed volume has a side length of 5 μm . The orientation accuracy in this study is estimated to be better than 0.2° . For analysis purposes, only the interior grains are used, with those adjacent to the edge of the volumes removed, as shown in figure 2(b). The remaining numbers of interior grains are 525, 351 and 343 for states t_0 , t_1 and t_2 .

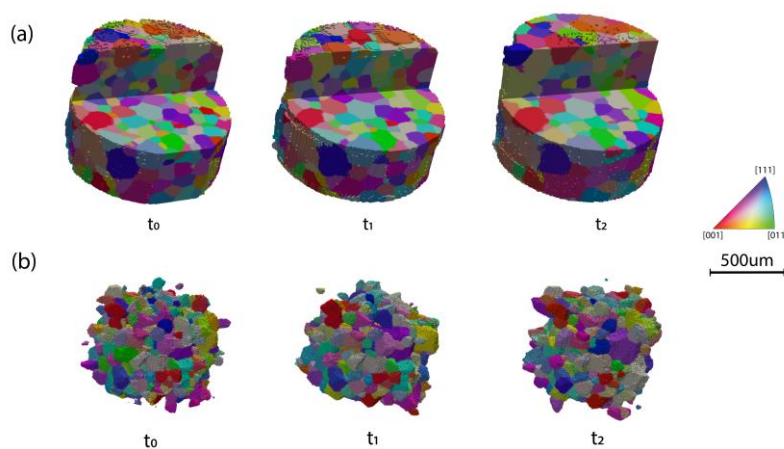


Figure 2. (a) The reconstructed 3D grain maps of the corresponding volume at the three temporal states t_0 , t_1 and t_2 . (b) The 3D grain maps with the grains adjacent to the edges of the volume removed. The colors of the grains are based on the orientation relative to the rotation axis.

The equivalent sphere diameter (ESD) is used to describe the grain size, as calculated from the volumes of the grains. The distributions of the grain size at the three temporal states are shown in figure 3. The size of the grains is distributed within a similar range after the two annealing treatments, and it is seen that grain growth primarily occurred during the 1st annealing treatment while the 2nd annealing treatment only resulted in minor changes in the grain size distributions. As revealed from the 2D slices at similar position within the reconstructed volumes (figure 4), the three temporal states have similar microstructure on the overall scale with only local variations.

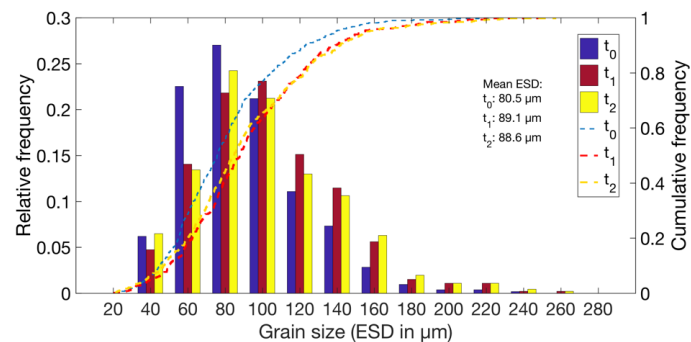


Figure 3. Distributions and cumulative frequency curves for the grain size of the three temporal states.

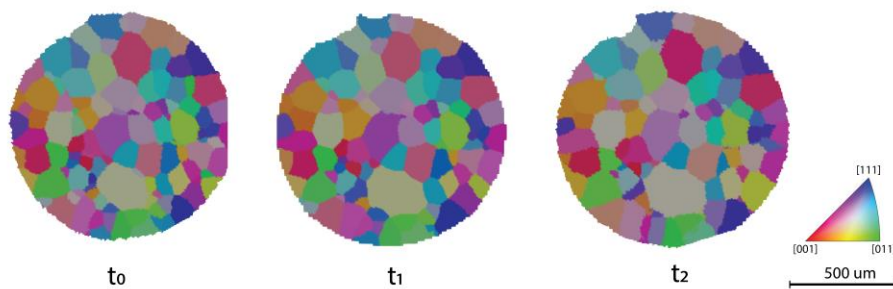


Figure 4. Slice from similar position in the reconstructed volume showing the microstructure of the sample at three temporal states. The colors of the grains are based on the orientation of the grain relative to the rotation axis.

Among the reconstructed 3D grains (excluding those grains adjacent to the volume edge) mapped in the three temporal states, 287 grains were found to have an exact match in orientation and reasonable match in center-of-mass positions. The grain growth behavior for these grains was characterized by the change in equivalent sphere diameter and it was found that only 18 grains had grown more than 10% in their equivalent sphere diameter after the two annealing treatments. The orientations of these growing grains, as well as their grain boundary misorientations to the neighboring grains, were analyzed. As shown in figure 5(a), the orientation of these growing grains (red dots) are almost randomly distributed, and the distribution of misorientation between the growing grains with their neighbors is similar with that of all the grains (figure 5b). No specific orientation correlation with the growing of grains is observed for the current sample.

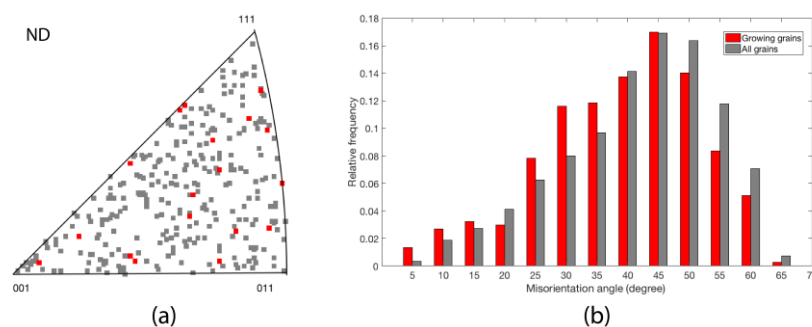


Figure 5. (a) Inverse pole figure of the sample rotation axis (ND for the hot-rolled bar). (b) Distribution of the misorientation angle between grains and their neighbors. The color scheme is the same for (a) and (b): red represents the grains that have grown more than 10% in the ESD and grey represents all the 287 grains.

4. Summary and Outlook

The paper presents a first 4D study of grain growth kinetics enabled by the use of laboratory X-ray diffraction contrast tomography (LabDCT). It is demonstrated that essential information for the study of grain growth, such as the shape, size and crystallographic orientation of the grains, can be obtained non-destructively at multiple temporal states. This methodology provides access to the necessary 4D experimental evidence which previously were not readily available. It is found that for the current Armco iron specimen, no substantial grain growth has occurred with current annealing treatment, and that for the small number of grains that have grown, there is no obvious correlation with their crystallographic orientation.

This work is ongoing and several aspects are suggested for future study:

- New experiments with more time steps should be conducted. The current study only includes two annealing treatments and is limited by the relative small amount of grain growth occurring during the annealing treatments given to the samples.
- Topological parameters such as the number of faces for individual grains should also be analyzed and correlated with the grain growth kinetics, with the results used to validate the existing theoretical models.

Acknowledgement

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